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(54) Title: POLISHING COMPOSITION AND METHOD

(57) Abstract: To provide a polishing composition which enables maintenance of excellent properties and high quality of the surface of a hard disk without lowering polishing rate during polishing of the surface, and which can provide a polished surface in which the amount of dub-off is considerably reduced as compared with that of a conventional level, a polishing composition containing water, a polishing material (particularly alumina), a polishing accelerator, and at least one of hydroxypropyl cellulose and hydroxyalkyl alkyl cellulose is provided.

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## DESCRIPTION

POLISHING COMPOSITION AND METHOD

## Technical Field

5 The present invention relates to a polishing composition which is employed for precise polishing of metal, plastic, or glass, particularly employed for precise finishing of the surface of an aluminum magnetic disk (hereinafter the disk will be referred to as a "hard disk") which is installed in a hard disk drive of a computer.

10 Background Art

In recent years, as high-performance computers have been developed and computers have been downsized, there has been demand for high-quality mirror-surface finishing in hard disks without surface defects, in accordance with an increase in recording density of the disks. In order to meet such demand for surface finishing, there have been attained a variety of technical developments on polishing compositions, polishing pads, polishing machines, and polishing techniques.

15 20

For example, Japanese Patent Application Laid-Open (kokai) No. 62-25187 discloses a polishing composition containing an inorganic salt, serving as a polishing accelerator, such as nickel nitrate or aluminum nitrate for increasing polishing rate. Japanese Patent Application Laid-Open (kokai) No. 2-84485 discloses a polishing composition which contains an organic acid such as gluconic acid or lactic acid, and a sodium salt thereof for increasing polishing rate, and which can provide a polished surface with reduced surface defects.

25 30

Japanese Patent Application Laid-Open (kokai) No. 7-216345 discloses a polishing composition which contains an organic acid, a molybdic acid salt, and alumina sol so as to attain a high polishing rate and a polished surface with reduced surface defects. These polishing compositions have been developed in order to maintain

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high rate of polishing a hard disk, reduce surface roughness and surface defects, and increase recording density.

5           Meanwhile, Japanese Patent Application Laid-Open  
(*kokai*) Nos. 5-2747 and 5-89459 disclose methods for  
reducing dub-off at the circumferential end of a hard  
disk and for increasing recording area, in order to  
increase recording capacity per hard disk. However,  
these publications do not disclose a polishing  
10       composition, although they disclose conditions for  
polishing. Japanese Patent Application Laid-Open (*kokai*)  
No. 1-263186 discloses a polishing composition containing  
triethanolamine carboxylic acid, triethanol  
hydrochloride, and aluminum stearate for reducing the  
15       amount of dub-off. However, since hard disks these days  
must meet very strict requirements concerning surface  
roughness, such a polishing composition cannot be  
directly applied to high-precision finishing of polished  
surfaces.

20           The polishing compositions disclosed in the above  
publications have been developed in order to enhance  
polishing rate, to reduce surface defects such as micro-  
pits and micro-protrusions as well as scratches on the  
surface of a hard disk for improvement of quality, and to  
25       reduce surface roughness for increasing recording  
density. Incidentally, there has been demand for  
increasing recording capacity in a hard disk of  
conventional size. In order to increase recording  
capacity of a hard disk, recording density per unit area  
30       in the disk must be increased. However, during polishing  
of a hard disk, the circumference of the disk is  
excessively polished to form a curve portion. Such an  
unavoidable curve portion is called "dub-off" or "roll-  
off," and a region containing dub-off in a hard disk  
35       cannot be employed for recording. If the amount of dub-  
off can be reduced as much as possible, recording  
capacity per hard disk can be increased. Therefore,

there has been demand for minimizing the amount of dub-off in a hard disk.

In view of the foregoing, an object of the present

invention is to provide a polishing composition and

method which can maintain polishing rate during polishing of the surface of a hard disk, maintain excellent

properties and high quality of the surface of the disk,

and provide a finished surface in which the amount of

dub-off is considerably reduced as compared with that of a conventional level.

Disclosure of the invention

The present invention provides a polishing

composition comprising water, a polishing material

(particularly alumina), a polishing accelerator, and at

least one of hydroxypropyl cellulose (hereinafter

abbreviated as "HPC") and hydroxyalkyl alkyl cellulose

(hereinafter abbreviated as "HRC"). The polishing

composition is mainly employed for polishing the surface

of a hard disk which is installed in a computer, but is

not limited thereto. Particularly, the composition can

provide a finished surface in which the amount of dub-off

is considerably reduced as compared with that of a

conventional level, while maintaining high polishing

rate, high surface quality, and excellent surface

roughness. The present invention also provides a method

for polishing a workpiece using such a polishing

composition.

Brief Description of Drawings

Fig. 1 is a schematic representation employed for

explanation of determination of the amount of dub-off, in

which

S: Curve in the vicinity of the circumferential end

of a disk, which is drawn by use of a surtrecorder

h: Perpendicular line brought into contact with the

circumferential end of a disk

A: Point on the curve which is 3,000  $\mu$ m from

perpendicular line h

B: Point on the curve which is 2,000  $\mu\text{m}$  from perpendicular line h

C: Point on a linear line passing through points A and B, which is 500  $\mu\text{m}$  from perpendicular line h

5 k: Perpendicular line passing through point C

D: Point at which perpendicular line k and curve S cross

t: Length between point C and point D (the amount of dub-off)

10 Best Modes for Carrying Out the Invention

An unavoidable phenomenon during polishing by use of free abrasive grains is occurrence of dub-off in a polished disk. The mechanism of dub-off is not necessarily clarified. However, through performing  
15 polishing operation over years, it has been found that when polishing rate is high, the amount of dub-off of a disk is reduced, but surface roughness generally increases and protrusions tend to be generated on the disk; and that when polishing rate is low, the amount of  
20 dub-off of a disk increases and pits tend to be generated on the disk. Meanwhile, it has been found that when a disk sinks deeply into a polishing pad, the amount of dub-off of the disk tends to increase. On the basis of these findings, extensive studies have been performed on  
25 a variety of additives, for example, in order to increase the viscosity of a polishing solution while the performance of a polishing material contained in the solution is maintained. The polishing composition of the present invention has been accomplished on the basis of  
30 the studies.

In the present invention, the amount of dub-off is determined as follows, as described with reference to Fig. 1.

35 As shown in Fig. 1, a circumferential portion of a polished hard disk is traced along the surface by use of a surf-corder to draw a curve S. A perpendicular line h

is drawn along the circumferential end of the curve S. Points on the curve S which are 3,000  $\mu\text{m}$  and 2,000  $\mu\text{m}$  from the perpendicular line h towards the center of the disk are assigned A and B, respectively. On a linear line passing the points A and B, a point which is 500  $\mu\text{m}$  from the perpendicular line h is assigned C. A perpendicular line k is drawn so as to pass the point C, and a point at which the perpendicular line k and the curve S cross is assigned D. The length t between the points C and D is determined as the amount of dub-off of the disk.

In order to confirm the effect of a thickener for reducing dub-off, a variety of polymers were evaluated in terms of water-solubility or other properties.

Consequently, it was found that when hydroxypropyl cellulose (HPC) or hydroxyalkyl alkyl cellulose such as hydroxypropyl methyl cellulose (HPMC), hydroxyethyl methyl cellulose (HEMC), or ethyl hydroxyethyl cellulose (EHEC), which has a more steric fiber structure among water-soluble cellulose derivatives, is added to a polishing composition, the polishing composition can provide an excellent polished surface with a small amount of dub-off while high polishing rate and high surface accuracy are maintained.

The mechanism of reduction in the amount of dub-off through the addition of HPC, HPMC, HEMC, or EHEC has not yet been elucidated, but the molecular structure or the type of end group of cellulose ether and thickening through the addition of the cellulose may be attributed to reduction in the amount of dub-off.

No particular limitation is imposed on the form of crystal structure, such as  $\alpha$ ,  $\theta$ , or  $\gamma$ , of alumina which is preferably employed as a polishing material in the present invention, but  $\alpha$ -alumina is more preferable in consideration of polishing rate. The particle size of alumina is determined according to the desired surface

roughness of a disk. The mean particle size of alumina is generally 0.02-5  $\mu\text{m}$ , preferably 0.1-3  $\mu\text{m}$ . The particle size distribution of alumina may be preferably as narrow as possible. The amount of alumina may be 1-30 wt.% on the basis of the entirety of a polishing composition, preferably 3-20 wt.%.

A polishing material which is employed in the present invention is not limited to alumina, and silica, titania, zirconia, or cerium oxide may be employed to obtain an effect similar to that of alumina. These polishing materials may be employed in combination.

The particle size and the amount of the polishing material which is employed may be determined in a manner similar to the case in which alumina is employed, but particle size and amount may be changed.

A polishing accelerator which may be employed in the present invention may be an organic acid or an inorganic acid salt. An organic acid is at least one species selected from the group consisting of malonic acid, succinic acid, adipic acid, lactic acid, malic acid, citric acid, glycine, aspartic acid, tartaric acid, gluconic acid, heptogluconic acid, iminodiacetic acid, and fumaric acid. Meanwhile, an inorganic acid salt is at least one species selected from the group consisting of sodium sulfate, magnesium sulfate, nickel sulfate, aluminum sulfate, ammonium sulfate, nickel nitrate, aluminum nitrate, ammonium nitrate, ferric nitrate, aluminum chloride, and nickel sulfamate. The amount of an organic acid or an inorganic acid salt which is incorporated into the polishing composition is preferably 0.003-10 wt.% on the basis of the entirety of the composition.

A polishing accelerator which may be employed in the present invention may be a combination of an organic acid and at least one of an organic acid salt and an inorganic acid salt. An organic acid is at least one species selected from the group consisting of malonic acid,

succinic acid, adipic acid, lactic acid, malic acid, citric acid, glycine, aspartic acid, tartaric acid, gluconic acid, heptogluconic acid, limnodiacetic acid, and fumaric acid. An organic acid salt which is employed in combination with the organic acid may be a potassium salt, sodium salt, or ammonium salt of the above organic acid. An inorganic acid salt which is employed in combination with the organic acid is at least one species selected from the group consisting of sodium sulfate, magnesium sulfate, nickel sulfate, aluminum sulfate, ammonium sulfate, nickel nitrate, aluminum nitrate, ammonium nitrate, ferric nitrate, aluminum chloride, and nickel sulfamate. When a combination of an organic acid and an organic acid salt or a combination of an organic acid and an inorganic acid salt is employed, the amount of the combination which is incorporated into the polishing composition is preferably 0.01-10 wt. % on the basis of the entirety of the composition. In this case, the amount of an organic acid is preferably at least 0.003 wt. % on the basis of the entirety of the composition.

When a combination of an organic acid and an organic acid salt is employed as a polishing accelerator, an organic acid and a salt of the same organic acid are preferably employed in combination so as to obtain excellent polishing properties of the composition.

The amount of HPC, HPMC, HEMC, or EHEC, which is employed in the polishing composition of the present invention singly or in combination, is 0.001-2 wt. % on the basis of the entirety of the composition. When the amount is very small, the effect of reducing dust is not obtained, whereas when the amount is very large, polishing rate decreases. The amount is preferably 0.01-1.0 wt. % on the basis of the entirety of the composition.

The aforementioned amount of each component in the polishing composition is the amount when the composition is employed for polishing a hard disk substrate.



Therefore, it is efficient that the polishing composition containing each component in an amount larger than that described above is produced and transported, and the composition is diluted upon use such that the amount of the component becomes as described above.

If necessary, in the polishing composition of the present invention, there may be employed, as an additive, alumina sol, a surfactant, a cleaning agent, a rust preventive, a preservative, a pH regulating agent, and a surface modification agent such as sulfamic acid or phosphoric acid which is known to exhibit the effect for reducing surface defects.

The polishing composition of the present invention preferably has a pH of 2-6.

#### Examples

The present invention will next be described in more detail by way of examples, which should not be construed as limiting the invention thereto.

Examples 1 through 15 are shown in Table 1, and Comparative Examples 1 through 6 are shown in Table 2.

#### (Preparation of polishing composition)

Aluminum hydroxide was heated at about 1,200°C in air in a firing furnace, to thereby obtain  $\alpha$ -alumina. The thus-obtained  $\alpha$ -alumina was crushed and subjected to wet-classification, thereby preparing alumina samples having mean particle sizes of 0.6  $\mu\text{m}$ , 0.7  $\mu\text{m}$ , and 1.0  $\mu\text{m}$ .

Subsequently, on the basis of compositions shown in Tables 1 and 2, water, alumina, a polishing accelerator, and HPC, HPMC, HEMC, or EHEC were weighed, incorporated, and mixed, to thereby prepare a polishing composition sample.

#### (Polishing conditions)

An NiP-plated aluminum disk (size: 3.5 inch) was employed as a workpiece to be polished. A polishing test and evaluation of the disk were carried out under the following conditions.

Polishing test conditions

Polishing test machine:

9B double-sided polishing machine  
(product of System Seiko K.K.)

Polishing pad: Poltex DG

Number of revolutions of surface plate:

upper surface plate 28 rpm,  
lower surface plate 45 rpm,  
sun gear 8 rpm

Feed rate of slurry: 100 ml/min.

Polishing time: 5 minutes

Operation pressure: 80 g/cm<sup>2</sup>

Evaluation of disk

Polishing rate: calculated by difference in  
weight before and after polishing

the disk

Quality of polished surface:

pits, protrusions, and scratches on disks  
were observed under a microscope, and  
rating "good" was assigned when the total  
number of pits is not more than 10 for  
both sides of five disks, the total number  
of protrusions is 0 for both sides of five  
disks, and the total number of scratches  
is not more than 5 for both sides of one  
disk

Amount of dub-off:

measured by use of a surfacorder (model:  
SE-30D, product of Kosaka Kenkyujo)  
(measured as shown in Fig. 1)

The results of polishing test of Examples and  
Comparative Examples are shown in Tables 1 and 2,  
respectively.

Table 1

Ex.	$\alpha$ -Alumina		Polishing accelerator				HPC/ HRRC	Evaluation of polishing		
	Particle size D <sub>50</sub>	Amount	Organic acid		Organic acid salt/ inorganic acid salt			Polish- ing rate	Surface defect	Amount of dub- off
			Type	%	Type	%				
1	$\mu\text{m}$ 0.7	6	Lactic acid	0.5	Sodium lactate	1.0	HPC 0.1	$\mu\text{m}/\text{min}$ 1.13	-	$\text{\AA}$ 300
2	0.6	6	Lactic acid	0.5	Sodium lactate	1.0	HPC 0.1	0.78	Good	650
3	0.7	6	Lactic acid	4.0	Sodium lactate	5.0	HPC 1.0	1.15	Good	350
4	1.0	6	Malic acid	0.7	Sodium malate	0.2	HPC 0.1	1.35	Good	100
5	0.7	6	Malic acid	0.7	Sodium malate	0.2	HPC 0.1	1.24	Good	450
6	0.6	6	Malic acid	0.7	Sodium malate	0.2	HPC 0.1	0.88	Good	600
7	0.7	6	Malic acid	0.7	Sodium malate	0.2	HPC 1.0	1.04	Good	450
8	0.7	6	Malic acid	5.0	Sodium malate	4.0	HPC 1.0	1.25	Good	500
9	0.7	6	Malic acid	0.7	Sodium malate	0.2	HPMC 0.1	1.22	Good	450
10	0.7	6	Malic acid	0.7	Sodium malate	0.2	HPMC 0.1	1.21	Good	500
11	0.7	6	Gluconic acid	0.5	Sodium gluconate	0.5	HPC 0.1	0.98	Good	400
12	0.6	6	Gluconic acid	0.5	Sodium gluconate	0.5	HPC 0.1	0.73	Good	500
13	0.7	6	Lactic acid	0.5	-	-	HPC 0.1	1.02	Good	350
14	0.7	6	Malic acid	0.7	Nickel sulfate	0.3	HPC 0.1	1.09	Good	450
15	0.7	6	-	-	Aluminum nitrate	1.0	HPC 0.1	1.13	Good	400

Table 2

Comp. Ex.	$\alpha$ -Alumina		Polishing		accelerator		HPC/ HRRC	Evaluation of polishing		
	Particle size D <sub>50</sub>	Amount	Organic acid	Type	Organic acid salt/inorganic acid salt	Type		Polishing rate	Surface defect	Amount of dub-off
1	0.7	6	Lactic acid	0.5	Sodium lactate	1.0	0	1.18	Good	800
2	1.0	6	Malic acid	0.7	Sodium malate	0.2	0	1.27	Good	1000
3	0.7	6	Malic acid	0.7	Sodium malate	0.2	0	1.18	Good	1400
4	0.6	6	Malic acid	0.7	Sodium malate	0.2	0	0.93	Good	1900
5	0.7	6	Gluconic acid	0.5	Sodium gluconate	0.5	0	1.08	Good	1600
6	0.6	6	Gluconic acid	0.5	Sodium gluconate	0.5	0	0.81	Good	2100

As is apparent from comparison of Table 1 with Table 2, when HPC, HPMC, or HEMC is incorporated into a polishing composition, the amount of dub-off is reduced; i.e., the composition is improved.

5       Industrial Applicability

As described above, the polishing composition of the present invention comprising water, alumina, a polishing accelerator, and at least one of HPC and HRRC enables maintenance of a predetermined polishing rate, surface accuracy, and mirror surface without surface defects, and  
10       can provide excellent polishing performance so as to reduce the amount of dub-off.

CLAIMS

1. A polishing composition comprising water, a polishing material, a polishing accelerator, and at least one of hydroxypropyl cellulose and hydroxyalkyl alkyl cellulose. 5
2. A polishing composition according to claim 1, wherein the polishing material is selected from among alumina, silica, titania, zirconia, and ceria.
3. A polishing composition according to claim 1, wherein the polishing material is alumina. 10
4. A polishing composition according to any one of claims 1, 2 and 3, wherein the polishing accelerator comprises an organic acid or an inorganic acid salt. 15
5. A polishing composition according to any one of claims 1, 2, and 3, wherein the polishing accelerator comprises an organic acid and at least one of an organic acid salt and an inorganic acid salt.
6. A polishing composition according to any one of claims 1 through 5, wherein the organic acid is at least one species selected from the group consisting of malonic acid, succinic acid, adipic acid, lactic acid, malic acid, citric acid, glycolic acid, tartaric acid, gluconic acid, heptogluconic acid, iminodiacetic acid, and fumaric acid. 20
7. A polishing composition according to any one of claims 4 through 6, wherein the inorganic acid salt is at least one species selected from the group consisting of sodium sulfate, magnesium sulfate, nickel sulfate, aluminum sulfate, ammonium sulfate, nickel nitrate, aluminum nitrate, ammonium nitrate, ferric nitrate, aluminum chloride, and nickel sulfamate. 30
8. A polishing composition according to claim 5, wherein the organic acid salt is a potassium salt, a sodium salt, or an ammonium salt of the organic acid as recited in claim 6. 35
9. A polishing composition according to any one of claims 1 through 8, wherein the amount of the polishing

accelerator is 0.01-10 wt.% on the basis of the entirety of the composition.

10. A polishing composition according to any one of claims 1 through 9, wherein the hydroxyalkyl alkyl  
5 cellulose is at least one species selected from the group consisting of hydroxypropyl methyl cellulose, hydroxyethyl methyl cellulose, and ethyl hydroxyethyl cellulose.

11. A polishing composition according to any one of  
10 claims 1 through 10, wherein the amount of hydroxypropyl cellulose and/or hydroxyalkyl alkyl cellulose is 0.001-2 wt.% on the basis of the entirety of the composition.

12. A method for a precision polishing, comprising  
15 polishing a workpiece with a polishing composition comprising water, a polishing material, a polishing accelerator, and at least one of hydroxypropyl cellulose and hydroxyalkyl alkyl cellulose.

13. A method according to claim 12, wherein said workpiece is an aluminum magnetic disk substrate.

20 14. A polishing composition according to claim 12 or 13, wherein the polishing material is selected from among alumina, silica, titania, zirconia, and ceria.

15. A polishing composition according to claim 12 or 13, wherein the polishing material is alumina.

25 16. A polishing composition according to any one of claims 12 through 14, wherein the polishing accelerator comprises an organic acid or an inorganic acid salt.

17. A polishing composition according to any one of  
30 claims 12 through 16, wherein the polishing accelerator comprises an organic acid and at least one of an organic acid salt and an inorganic acid salt.

18. A polishing composition according to any one of  
35 claims 12 through 17, wherein the organic acid is at least one species selected from the group consisting of malonic acid, succinic acid, adipic acid, lactic acid, malic acid, citric acid, glycine, aspartic acid, tartaric acid, gluconic acid, heptogluconic acid, iminodiacetic

acid, and fumaric acid.

19. A polishing composition according to any one of claims 16 through 18, wherein the inorganic acid salt is at least one species selected from the group consisting of sodium sulfate, magnesium sulfate, nickel sulfate, aluminum sulfate, ammonium sulfate, nickel nitrate, aluminum nitrate, ammonium nitrate, ferric nitrate, aluminum chloride, and nickel sulfamate.

20. A polishing composition according to claim 17, wherein the organic acid salt is a potassium salt, a sodium salt, or an ammonium salt of the organic acid as recited in claim 18.

21. A polishing composition according to any one of claims 12 through 20, wherein the amount of the polishing accelerator is 0.01-10 wt. % on the basis of the entirety of the composition.

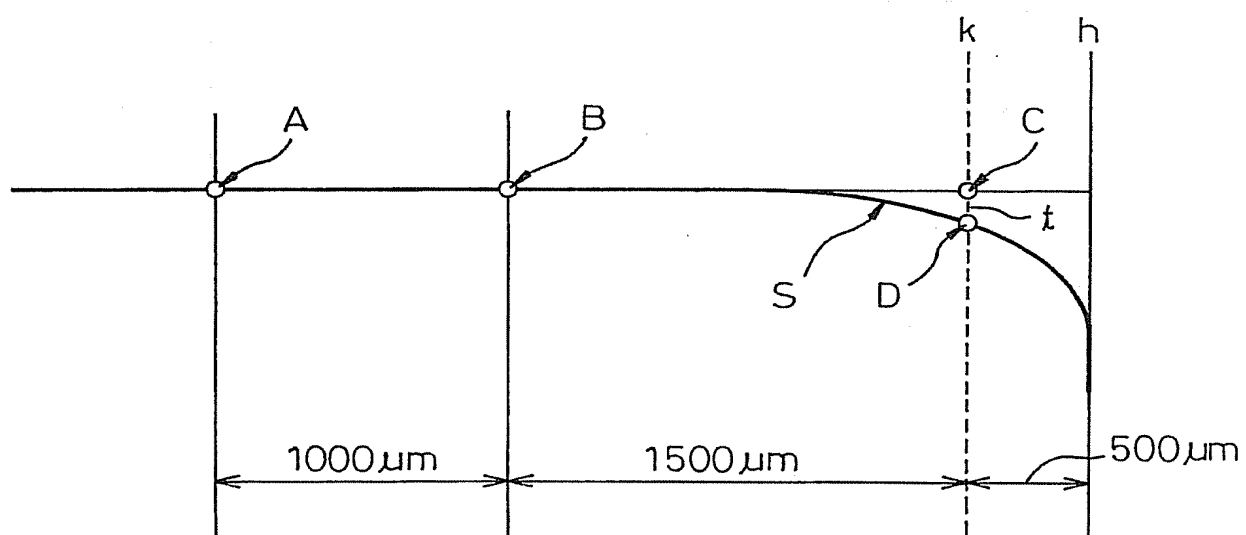
22. A polishing composition according to any one of claims 12 through 21, wherein the hydroxyalkyl alkyl cellulose is at least one species selected from the group consisting of hydroxypropyl methyl cellulose, hydroxyethyl methyl cellulose, and ethyl hydroxyethyl cellulose.

23. A polishing composition according to any one of claims 12 through 22, wherein the amount of hydroxypropyl cellulose and/or hydroxyalkyl alkyl cellulose is 0.001-2 wt. % on the basis of the entirety of the composition.



$\frac{1}{1}$ 

Fig.1



# INTERNATIONAL SEARCH REPORT

Int'l. Application No  
PCT/JP 00/06805

A. CLASSIFICATION OF SUBJECT MATTER  
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According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)  
IPC 7 C09G C09K C23F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

WPI Data, PAJ, EPO-Internal

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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P, X	EP 0 967 260 A (FUJIMI INC.) 29 December 1999 (1999-12-29) page 3, line 43-50 page 4, line 2-4 page 4, line 47-54 EP 0 842 997 A (NISSAN CHEMICAL IND.) 20 May 1998 (1998-05-20) abstract page 2, paragraph 1 page 3, line 34-47 --- -/-	1-4, 9-11, 14-16, 21-23  1-4, 9, 10, 12-16, 19, 21, 22
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<input checked="" type="checkbox"/> Further documents are listed in the continuation of box C.	<input checked="" type="checkbox"/> Patent family members are listed in annex.
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Date of the actual completion of the international search	2 January 2001
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Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl. Fax: (+31-70) 340-3016	Authorized officer Girard, Y
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# INTERNATIONAL SEARCH REPORT

International Application No

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## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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